Pat.Appln.Nr 09/872,990   Docket 437-01US

Remarks,
responsive to O/A dated 11 December 2002

1.   Claim 1 is amended by the incorporation therein of claim 6.

Since claim 6 excludes the claim 7 condition, we have deleted claim 7.

2.   In view of the amendments, the following is a response to the 35 USC 103 rejection of claim 6, rather than to the rejection of claim 1.

3.   The PTO position is that the skilled person would find it obvious to modify the procedure disclosed by Christy, by adding a step as disclosed by Burnham, and a step as disclosed by McMahon, to the steps as disclosed by Christy.

The examiner rejects claim 1 under 35 USC 103, on the basis that the skilled expert would find it obvious to add the physical shearing step as disclosed by McMahon to the raised pH and temperature step as disclosed by Christy.

We ask that this rejection be reconsidered and withdrawn, for the following reasons.

4.   It is our position that combining McMahon's shearing with Christy's raising of the pH and temperature is inventive. We feel the skilled person would not find it obvious to combine the steps as now recited in (amended) claim 1. We will show that the benefits arising from the fact of the combination of the steps is more than would be expected from a mere aggregation of the steps. That being so, inventiveness is demonstrated.

5.   As mentioned in our specification, one of our objectives in this case was to reduce viscosity of the sludge, to enable the sludge to be pumped. As a generality, ten-percent-solid sludges are so thick and viscous as to be almost un-pumpable. Persons concerned with moving or handling sludge prefer it to be at the five-percent-solids level, or less. Against that, however, even a ten-percent sludge is ninety percent water, and the transport of that is economically wasteful. We were seeking for a way of reducing the viscosity of a high-solids-content sludge.

As mentioned in the specification, we are also concerned with elimination of pathogens from the sludge, and also with creating conditions in the sludge which will discourage re-growth of whatever pathogens might remain. Our objective was to seek to reduce the viscosity of a high-solids-content sludge, so the sludge can be pumped, but at the same time to eliminate /inhibit pathogens.

6. Combination of shearing the sludge with raising the pH of the sludge

6.1 We present two lines of argument, to the effect that this combination provides a previously-unrealised synergy.

6.A.1 It is true that vigorously shearing a substance can sometimes make the substance less viscous. As an everyday example, a solid or almost-solid food item can be transformed into a liquid, or at least into a paste, in a kitchen blender (homogeniser). Thus, we may accept the notion, for the moment, that subjecting a sludge to shearing will serve to make the sludge less viscous.

(We point out, however, that some substances become more viscous when sheared. In some cases, the action of tearing open bio-cells, during shearing, can release large organic molecules, which can have the effect of increasing viscosity. There is no teaching that any and every sludge will inevitably and always be made less viscous upon being subjected to any and every manner of shearing.)

It is known that increasing the pH of a sludge to e.g eleven or twelve will make the sludge less viscous. And it is known that this high alkalinity step reduces the viscosity of the sludge in the absence of any other viscosity-changing step.

But the prior art does not disclose that the step of shearing sludge has previously been combined with the step of raising the pH of the sludge. The PTO position is that it would obviously occur to the skilled person to combine those steps, e.g for the purpose of aggregating their viscosity-reducing effects.

6.A.2 However, we have recognised that, by combining the step of shearing the sludge with the step of raising the pH, we can produce beneficial effects that are much greater than would be expected by merely aggregating the separate effects of the two steps. The whole is greater than the sum of the parts; and where that is so, inventiveness is thereby demonstrated.

6.A.2 Vigorous shearing of sludge means the sludge becomes homogenised. Vigorous shearing means there are no (or fewer) clumps of solid material in the sludge. This is important because sludge tends naturally not to be homogeneous, whereby what can happen, when carrying out the step of raising the pH (which is done by adding pH-raising chemicals) of the sludge, is that the material in the middle of a solid clump can still be at a low pH, even though the sludge as a whole is measured as having a high pH. The time it takes for a body of sludge to reach a high pH throughout the body is much reduced if the sludge is sheared.

6.A.3 Stirring the sludge, if done while raising the pH, would not be very effective, i.e mere stirring would not be expected to reduce clumping in sludge. But shearing physically tears the material. That degree of violence is very effective to break up the clumps. Sludge that has been sheared is homogenised to a much greater degree than sludge that has been merely stirred.

So, even if shearing, by itself, had <u>no</u> effect to reduce viscosity, combining shearing with raising the pH enables the viscosity-reducing effects attributable to raising the pH to be much enhanced.

Just that fact is sufficient to demonstrate the inventiveness of combining the step of raising the pH with the step of shearing the sludge.

6.B.1. But shearing, per se, does also serve to reduce the viscosity of the sludge. Shearing breaks open the cells of the bio-solid materials, releasing the water locked up in the cells, and now this "free" water reduces the viscosity. It may be noted that, since shearing reduces viscosity, therefore the energy needed to carry out the shearing becomes less as shearing progresses. The greater the viscosity of the sludge when the shearing step is carried out, the more robust the shearing apparatus needs to be, and the more energy is needed. Raising the pH of the sludge means that the viscosity is already a little reduced, when the shearing is carried out. So, combining the shearing step with the pH-raising step serves to reduce the resources needed to carry out the shearing step. This would be true even if the designer were planning to reduce the viscosity of the sludge by shearing alone.

Again, just that fact is sufficient to demonstrate the inventiveness of combining the step of raising the pH with the step of shearing the sludge.

6.2.   The designer's reason for wanting to raise the pH of the sludge might be, not primarily to lower the viscosity of the sludge, but rather to enhance the pathogen-removal effectiveness of raising the pH. The points as mentioned above, that applying a shearing step serves to speed up what would be a slow penetration of the pH-raising chemicals into and throughout the whole body of sludge, apply whatever the designer's motives for seeking a raised pH.

7.   Combination of shearing the sludge with raising its temperature

7.1   Here, we provide three lines of argument, to the effect that this combination provides a previously-unrealised synergy.

7.A.1   As mentioned above, we will accept the notion, for the moment, that subjecting a sludge to shearing will serve to make the sludge less viscous.

Equally, while some substances become thinner (less viscous) at higher temperatures, it is certainly true that some other substances become thicker as they get hotter. But again, we will accept the notion, for the moment, that raising the temperature of a sludge will serve to make the sludge less viscous.

But the prior art does not disclose that the step of shearing sludge has previously been combined with the step of raising the temperature of the sludge. The PTO position is that it would obviously occur to the skilled person to combine those steps, e.g for the purpose of aggregating their pathogen-elimination effects.

Paralleling our comments in section 6 above, we have recognised that, by combining the step of shearing the sludge with the step of raising the temperature, we can produce beneficial effects that are much greater than would be expected by merely aggregating the separate effects of the two steps. The whole is greater than the sum of the parts; and where that is so, inventiveness is thereby demonstrated.

7.A.2 The wide use of the traditional combination of 70°C for 30 minutes, for pasteurization of sludge, has been commented upon in the prior art. For example, Havelaar states: "Laboratory data on thermal resistance of pathogens would suggest that 60°C for the same time would be sufficient but there are several reasons for choosing a more rigid temperature regime."

7.A.3 We have recognised that many of these "reasons" that inhibit the beneficial effects of temperature are addressed when the sludge is subjected to a shearing step.

Shearing the sludge gives it a more homogeneous texture. Large clumps of solid organic matter may protect the pathogens against the action of heat; and this is especially so if the pathogens are absorbed in e.g fat globules, into which heat will penetrate slowly. The more homogeneous the sludge material, the less possible it is for the pathogens to "hide" from the high temperatures, within the material.

Thus, the fact that the sludge has been subjected to a shearing step serves to make the step of raising the temperature more effective to eliminate pathogens. Just that fact is sufficient to demonstrate the inventiveness of combining the step of raising the temperature with the step of shearing the sludge.

7.B.1. In addition, an on-going shearing of the sludge, while the sludge is at high temperature, serves to enhance the effectiveness of applying the high temperature. On-going shearing has the effect of minimising temperature gradients within the body of sludge, which has the effect of maximising the rate at which heat can be transferred into the body of sludge. The more vigorously the sludge is sheared, the more rapidly heat will permeate and create an evenly high temperature throughout the whole body of the sludge.

Thus, the fact that the sludge is being subjected to an on-going shearing step serves to make the step of applying heat to the sludge more rapidly effective to produce high temperature evenly throughout the sludge. Again, just that fact is sufficient to demonstrate the

inventiveness of combining the step of raising the temperature with the step of shearing the sludge.

7.C.1. There is also another manner in which combining shearing with high temperature is beneficial, as follows. Vigorous shearing is effective to break open the cells of the bio-solid material that is a main constituent of the sludge. Shearing releases some of the water that was bound up in the cells now as "free" water. (In the absence of some other effects that might arise when cells are broken open) this released liquid water serves to reduce the viscosity of the body of sludge. It has been noted in the prior art (Fellows) that the higher the water content of a medium the higher the thermal death rate at a given temperature. We have recognised that this is still true even when the increased water activity occurs as a result of tearing the cells open.

Thus, the action of shearing the sludge serves to make temperature a more powerful agent for eliminating the pathogens. Again, just that fact is sufficient to demonstrate the inventiveness of combining the step of raising the temperature with the step of shearing the sludge.

8. Christy discloses treating sludge by raising its pH and raising its temperature. McMahon discloses the step of shearing to reduce viscosity. We feel the points made in section 6 above are sufficient to demonstrate that the effect of combining the shearing step with raising the pH is much greater than would be expected if the combined effect were merely an aggregate of the separate effects of those steps.

Furthermore, we fell the points made in section 7 above are sufficient to demonstrate that the effect of combining the shearing step with raising the temperature is much greater than would be expected if the combined effect were merely an aggregate of the separate effects of those steps.

Taking the two things together is doubly demonstrative of the inventiveness of claim 1.

9. The above is sufficient to demonstrate inventiveness. Therefore, we do not, in this response, need to contend that there is a separately synergistic effect arising from combining Christy's step of treating sludge by raising its pH and raising its temperature with Burnham's step of adding salt. Nor that there is a separately synergistic effect arising from adding Burnham's step of adding salt to the combination of Christy's raised pH and temperature with McMahon's shearing. However, this should not be construed as a denial of such effects.

10. New claims 29,30 bring out some aspects of the invention we now wish to emphasise, in the light of the points made herein.

11.   We re-present claims 20-23. In fact, these are simple product-by-process claims, derived from claim 1. Clearly, these claims can stand in this application.

12.   We ask that the rejection be reviewed, and withdrawn, and we look forward now to receiving a Notice of Allowance.

Anthony Asquith

References:
- Havelaar - 1984 - Paper entitled *'Sludge disinfection-an overview of methods and their effectiveness'* - copy enclosed (8 sheets)
- Fellows - 2000 - *Food Processing Technology, Principles and Practice, 2nd Ed.* CRC. Boca Raton, p43) - copy enclosed (2 sheets)

HAVELAAR.

Table of Contents

CHAPTER 2

Sludge disinfection—an overview of methods and their effectiveness

A. H. Havelaar

2.1 INTRODUCTION

Sewage sludge contains a variety of pathogens, often in numbers exceeding those in raw sewage. Thus, the use of untreated (raw) sewage sludge as a fertilizer in agriculture or horticulture presents a health risk to man, animals and plants.

It is now commonly accepted that raw sewage or sludge should not be used for the production of crops that are consumed by humans raw, or more strictly, that are brought into the kitchen raw. With regard to health risks connected with other agricultural uses of sewage sludge, there is little or no agreement however. Consequently, guidelines or legal requirements concerning the use of sludge in agriculture, may vary widely between different countries. There are several factors that may explain the divergent opinions. Regional factors like climate, type of animal husbandry or other agricultural practices, availability of land, infection rates among the human population and other social, political and economic factors may explain the differences between guidelines in different parts of Europe.

The epidemiological data concerning health hazards associated with the agricultural use of sewage sludge are poor. Some evidence has been obtained for the transmission of salmonellosis or cysticercosis to grazing cattle, but other routes of transmission exist and no definite conclusions can yet be drawn. Pending further epidemiological studies, decisions will have to be made mainly based on common sense, and usually a certain margin of safety will be allowed. This leads to the conclusion that in certain cases, the destruction of pathogens will be considered necessary.

This chapter will briefly discuss a number of sludge treatment processes that may lead to a significant reduction of pathogens. These will include processes with disinfection as the primary aim, like pasteurisation or irradiation, but also processes that are carried out mainly for other purposes such as stabilisation or conditioning before dewatering. If used properly, these latter processes may also lead to a product that is fully acceptable from a hygienic point of view. The processes to be discussed are the following:

- pasteurisation
- irradiation
- aerobic thermophilic stabilisation
- composting
- lime-treatment.

After a review of the disinfecting effect of these processes, an overview will be given of their present use in countries participating in the EEC project COST 68 ter.

2.2 MEASURING AND DEFINING DISINFECTION OF SLUDGE

Whether sludge is safe to use depends strongly on the type of application. Obviously, there is no need for complete sterilisation of the sludge. The objective should be to kill pathogens that could potentially cause a health hazard in a given application. Thus, in this chapter the disinfecting action of certain processes will be discussed primarily by considering data with regard to pathogenic micro-organisms. According to Working Party 3 of the EEC Concerted Action Committee COST 68 ter, sludge can be regarded as 'sanitized' if salmonellae and eggs of Taenia spp. and Ascaris spp. are completely destroyed (or at least rendered non-infective in the case of parasitic eggs). This definition implies that certain other pathogens (for example the more resistant viruses) may survive, but their presence is not normally considered a health risk in connection with general application on pastures, parks or playgrounds.

Unfortunately, there are but few data on the survival of relevant pathogens in sludge treatment. More data are available on the so-called indicator-organisms (coliforms, streptococci, total counts etc.) and where appropriate, these will also be used to assess the germicidal capacity of a given process.

2.3 PASTEURISATION

Pasteurisation is a process by which the sludge is heated to a high temperature for a relatively short time, for example 70 °C for 30 minutes. The process has been developed on a technical scale primarily in Switzerland, where the main goal is to eliminate salmonellae.

Pasteurisation does not reduce the putrescibility of sludge, so it has to be combined with some sort of stabilisation, usually anaerobic mesophilic digestion. Originally, the digestion step was carried our prior to the pasteurisation process. The pasteurised sludge was found to be highly susceptible to regrowth of entero-bacteria including Salmonella. It was not unusual to find higher numbers of enterobacteria in the pasteurised sludge after storage than in the raw sludge.

[1,2]. Attempts have been made to identify and correct the critical hazard points: macerating of sludge, separate pipework for raw and pasteurised sludge, filtration of air-vents, cooling water of drinking water quality, no connections (with possibly leaking valves) between pasteurised and non-pasteurised sludge, better control by personnel closed storage tanks etc. Nevertheless, the final product was invariably found to contain salmonellae and enterobacteria in high numbers. The theoretical basis for the susceptibility of pasteurisated sludge to regrowth is unknown. It is suggested that pasteurisation reduces the level of a competitive flora and will break up large organic molecules into easily assimilable compounds. Both factors may stimulate regrowth of enterobacteria that have survived the pasteurisation process in low numbers, or that were introduced by slight recontamination. It must be pointed out that regrowth is only a problem with certain bacterial species. Other pathogens like parasitic eggs, protozoan cysts and human or animal viruses are unable to grow outside the human or animal host.

The regrowth problem led to a temporary relaxation of the Swiss ordinance for sludge disinfection and research was encouraged to overcome this problem. This eventually led to the so-called pre-pasteurised concept, that is raw sludge is pasteurised and then fed into a digester. By using specially developed sludge/sludge heat exchangers the process can be carried out with about the same energy consumption as that normally required for the mesophilic digestion process [3]. In addition, the pre-pasteurised sludge was found to have a better dewatering capacity.

Swiss experience has demonstrated that if pre-pasteurised sludge is fed into a digester, no regrowth of enterobacteria occurs. In fact, if the sludge in the digester was initially contaminated, a sharp decrease of enterobacteria will be found and numbers will be reduced below detectable levels in a few weeks. Upon accidental introduction of contaminated sludge, a temporary rise in bacterial numbers is observed, but these will rapidly be reduced again. Apparently, the enterobacteria are not able to compete with the anaerobic flora in a digester and thus will be reduced in number.

Results from pilot-plant experiments in Germany [4] confirm the greater stability of pre-pasteurised sludge, although there were some interesting differences with the Swiss data. Raw sludge usually contained 10^7 to 10^8 coliforms/ml (in sludge, coliforms and enterobacteria may be regarded as roughly equivalent). After anaerobic mesophilic digestion, the coliform level was reduced to 10^4 to 10^5/ml. Post-pasteurisation reduced coliforms further to 0 to 10^2/ml but after storage under 'open-air' conditions, a rise to 10^6 to 10^7 was observed. Pre-pasteurisation of raw sludge also reduced coliforms to 0 to 10^1/ml and after subsequent digestion 10^3 to 10^4/ml were found. Upon storage, no further increase was found. Salmonella-levels were very low throughout all stages of all processes. Artificially introduced S. senftenberg was always killed during pasteurisation

From a microbiological point of view, the question which time-temperature combination should be chosen is an interesting one. Given that there is little heat-loss in certain types of sludge/sludge heat exchangers [3], the question is less relevant from an operational point of view. Reduction of the temperature of the pasteurisation process would lead to only slightly lower energy losses, and there seems to be relatively little merit in questioning the now widely applied but arbitrarily chosen combination of 70 °C for 30 minutes. Laboratory data on thermal resistance of pathogens in sludge would suggest that 60 °C for the same time would be sufficient but there are several reasons for choosing a more rigid temperature regime.

- a safety margin is required to account for scaling up effects, inhomogeneity etc;
- the large amount of solid organic matter may protect the pathogens against the action of heat; this is especially so if they are absorbed in aggregates (fat globules) into which heat will penetrate slowly;
- experiments on a technical scale have demonstrated that 60 °C is just marginal; 65 to 70 °C is necessary for a reliable kill.

There is little doubt that the pasteurisation process as presently applied is highly efficient for killing vegetative bacterial cells, parasitic ova and many viruses, although data on full-scale plants at present are restricted to enterobacteria and salmonellae. No reduction is to be expected for the more heat-tolerant forms of life, for example certain viruses (bovine parvo) and bacterial spores. In addition, Strauch and Berg [5] have shown that 30 minutes at 60 °C 65 °C or 70 °C is sufficient to kill ova of Ascaris lumbricoides in a post-pasteurisation pilot-plant.

In the Swiss full-scale plants, the sludge is heated by an external source. In the Federal Republic of Germany and in the United Kingdom, a different heating technique is being developed. This so-called submerged combustion process will be discussed later in this book by Kidson and Ray (Chapter 21).

2.4 IRRADIATION

Practical experience with the irradiation of sewage sludge in Europe is limited to one plant in the Federal Republic of Germany (Geiselbullach near Munich). A ^{60}Co-source is used to give a radiation dose of 300 krad. Recently, Suess et al. [6] have summarised the data on the performance of this plant. Typically, total counts are reduced by 2 log units; salmonellae and other enterobacteria (in culture medium in containers) by 5 to 6 log units. Faecal streptococci were reduced by only 2 log units. Similar data were reported earlier by a group of Purdue University, USA. Eggs of Ascaris suum were still viable but not capable of embryonation after irradiation. According to Sauer [8], the radioresistance of A. suum eggs depends strongly on the stage of development of the eggs

Freshly harvested eggs were most resistant, requiring 630 krad (from a ^{137}Cs-source) for a complete kill. If the eggs were left to hatch in well-aerated water at 26 to 30 °C, the radioresistance dropped steadily and was about 90 krad at the stage of full embryonation. Free embryos were highly sensitive (9 krad).

Little is known about the problem of recontamination of irradiated sludge. Regrowth of *Enterobacter* spp. but not of *E. coli*, has been observed (Suess, personal communication). From an operational point of view, it is interesting to note that irradiated sludge shows better dewatering abilities than non-irradiated sludge.

The radiation dose required for disinfection can be reduced considerably by using the synergistic action of heat. This principle is applied in the so-called thermoradiation process developed by Sandia Laboratories, Albuquerque USA. For instance, embryonation of *Ascaris lumbricoides* ova is not inhibited by heating for two hours at 47 °C. At 20 °C, a radiation dose of 80 krad is required to reduce embryonation with 3 log-units. At 47 °C, the same effect is reached by a dose of only 40 krad [9].

2.5 AEROBIC THERMOPHILIC STABILISATION

The aerobic thermophilic stabilisation process has, as the name implies, been designed primarily for stabilisation. The high aeration rate used in this process initiates biological processes of such intensity that the heat generated by these processes results in temperatures at a disinfecting level. Basically, two concepts can be distinguished: aerating with pure oxygen or with air. If pure oxygen is supplied, the temperatures achieved usually surpass 60 °C and figures as high as 80 °C have been reported. Obviously, the disinfecting effect of this process will be at least equivalent to that of pasteurisation, and often quite better results may be expected because of longer retention times.

If the oxygen supplied comes from air, the temperature achieved can be between 40 °C and 60 °C, depending primarily on sludge solid content, retention time and the efficiency of the aerator. In this temperature range, death rates are much reduced, so that longer times are necessary for pathogen inactivation. The time-temperature relationships to reach a certain effect are not simple, straightforward nor can they be universally applied. The actual death rate of a pathogen will also depend on sludge characteristics like pH, solid content, detergents etc. and on the design of the digester. Additionally, tailing effects may be observed, that is after an initial relatively rapid kill, a proportion of the population will survive for a longer time [16]. According to Strauch [11] three or more hours at least 50 °C are necessary to obtain a reliable kill of *Salmonella, Ascaris suum* eggs or bovine enterovirus. These data are obtained by introducing pathogens into the stabilisation tanks in some kind of germ carrier, for example attached to leather strips that were contained in porous containers ("tea-eggs"). This design

implies that the residence times of the experimentally introduced pathogens are exactly known.

In the practical situation, however, the pathogens will be freely suspended in sludge continuously mixed through the digester. This implies that reductions as expected on the basis of a time-temperature relationship may not be reached in full scale plants because of short-circuiting. In fact, the mixing characteristics of the process seem to influence the final result more than the time-temperature combination. According to Berg and Berman [12] aerobically digested sludge at ca. 49 °C with an average residence time of 20 days still contained substantial numbers of indicator bacteria and enteric viruses. Mean reduction values were (in log units): total coliforms 5.5; faecal coliforms 4.4; faecal streptococci 3.4 and enteric viruses >2.6. In this study, feeding fresh sludge was carried out just prior to withdrawal of digested sludge. Nevertheless, all groups of micro-organisms studied were removed to a greater extent by aerobic thermophilic digestion than by mesophilic anaerobic digestion (20 days at 35 °C).

Kabrick and Jewell [13] reported extensive studies of aerobic thermophilic stabilisation in a digester, typically operating at 45 to 55 °C. The retention time was usually 20 to 30 days and a time lapse of 12 to 24 hours was applied between feeding and drawing. In the digested sludge, salmonellae and enteric viruses were usually reduced below detectable levels. Faecal coliforms and faecal streptococci were reduced by ca. 3.5 and 2.5 log units, respectively. Occasionally, viable parasitic eggs were demonstrated in the digested sludge. As in the former study, the pathogen removal of the aerobic thermophilic process was better than that of a mesophilic anaerobic digestion.

2.6 COMPOSTING

Composting is a stabilisation process that relies upon the aerobic breakdown of organic matter by thermophilic bacteria. The sludge is mixed with bulking agents that serve to increase porosity for good aeration, to reduce moisture content, and to improve the C:N-ratio [14]. Frequently, all three functions are combined in one product, for example straw, wood chips or household refuse. Non-degradable materials like plastics can also be used. Many variables may be introduced in the design and operation of composting processes. It is therefore difficult to give general data on the expected sanitising effect. Basically, two processes can be distinguished: composting in windrows or composting in bioreactors.

For windrow composting in its most elementary form, sludge is mixed with a bulking agent and shaped into piles. In order to obtain a hygienically safe product, it is important that all the sludge reaches a critical temperature for a certain period of time. This can be obtained by regular turning of heaps, for example once a week. Whether the end product will be safe depends on climate and other factors. Strauch et al [15] demonstrated that salmonellae will

be completely killed in summertime within 6 to 7 weeks, but in wintertime a complete reduction was only found in the centre of the windrows.

A much more elegant design has been developed by a group at the US Department of Agriculture, Beltsville, USA [16]. In this system, the so-called Beltsville Aerated Pile Method, the mixture of sludge and bulking agent is aerated by suction of air through perforated pipes under the windrows. The exhaust air is filtered through matured compost to prevent odour problems. This also leads to more uniform temperature profiles in the composting material and thus to a hygienically more safe product. According to Burge et al. [17] the system performs well in all seasons. Coliforms and salmonellae are reduced below detection level, that is by more than 7 and 2 log units respectively within 10 days.

The heat-resistant bacterial virus f2 artifically inoculated to ca. 10^7 pfu/g was removed within 13 days in most parts of the heap. At the outer edge, survival was longer (> 20 days). Bertoldi et al. [18] reported reduction of even the highly resistant spores of sulphite-reducing clostridia by 3 log units.

An appropriate control of temperature during composting is possible in so-called bioreactors as developed mainly in the Federal Republic of Germany. The fate of pathogens in bioreactors has been studied extensively by Strauch and co-workers [19, 20]. According to this group, a hygienically safe product will be obtained if a temperature of 60 °C is maintained for at least 24 hours (personal communication). Thus, pathogens resist high temperatures better in the composting process than in the pasteurisation process. This may be explained by several factors:

• reduced moisture content diminishes death rate
• less rapid heat penetration into solid particles
• inhomogeneities in the composting material.

According to several authors, some bacterial pathogens are able to regrow in composted material. Burge et al. [17] state that salmonellae regrow to a small extent but are unable to compete well with other bacteria. According to Löfgren et al. [21], strong regrowth of Klebsiella spp. may occur in later stages of composting, leading to a sharp increase of thermotolerant coliform counts.

2.7 LIME-TREATMENT

Calcium hydroxide (slaked lime) is frequently added to liquid sludge as a conditioning step before dewatering in filter-presses. This will result in an increase of pH that may reach values between 9 and 13, depending on lime-dose and sludge characteristics.

Vegetative bacterial cells (for example coliforms or salmonellae) are rapidly destroyed at pH values above 9 to 10. In sludge, however, their destruction is hindered by slow penetration of lime into aggregates. According to Strauch and Berg [22], pH should rise to above 11.5 in order to obtain a reliable kill. At these pH-values, most viruses will also be rapidly destroyed (Lund, personal communication). Virus destruction is caused not only by direct effects of high pH but also by release of free ammonia at pH-values around 12 [23].

Parasitic eggs seem to be rather insensitive to high pH. The viability and infectivity of thick-shelled ova of Ascaris suum was only slightly reduced by a pH of 12 for as much as 48 hours. [22, 24].

A complete destruction of even Ascaris eggs may be obtained by the addition of quicklime to dewatered sludge. Temperatures will rise as high as 70 to 80°C and a rapid kill of pathogens is observed [25]. Incomplete penetration of aggregates is no problem in the quicklime-process because the main lethal factor is the action of heat.

If limed sludge is stored in stockpiles, care should be taken to prevent regrowth of bacteria, including salmonellae. If the lime dose was sufficient to maintain a pH-level of 10 or more at all times, no problems should arise.

2.8 DISINFECTION OF SEWAGE SLUDGE IN MEMBER COUNTRIES OF THE COST 68 TER PROJECT

In the framework of the above mentioned project, an enquiry was held among member states to assess to what extent the processes discussed in this chapter are used in actual practice [26]. The results of this enquiry are summarised in Figure 2.1. It can be seen that important regional differences do occur. Lime treatment and composting are particularly carried out in the Scandinavian countries. It can also be seen that the mass of sludge that is purposefully disinfected is very small (17,000 tonnes dry matter/year or 0.3% of the total production).

Pasteurisation and irradiation are mainly carried out in Switzerland and the Federal Republic of Germany. In the next few years, a rise of the disinfected sludge mass in these countries may be expected because of the recent publication of relatively strict ordinances.

2.9 SUMMARY

The main features of the processes described above are outlined in the summary table. Obviously, the table is a simplification of the actual data, so that it can only be used for a general comparison of the processes. For more specific information the text or the original references should be consulted.
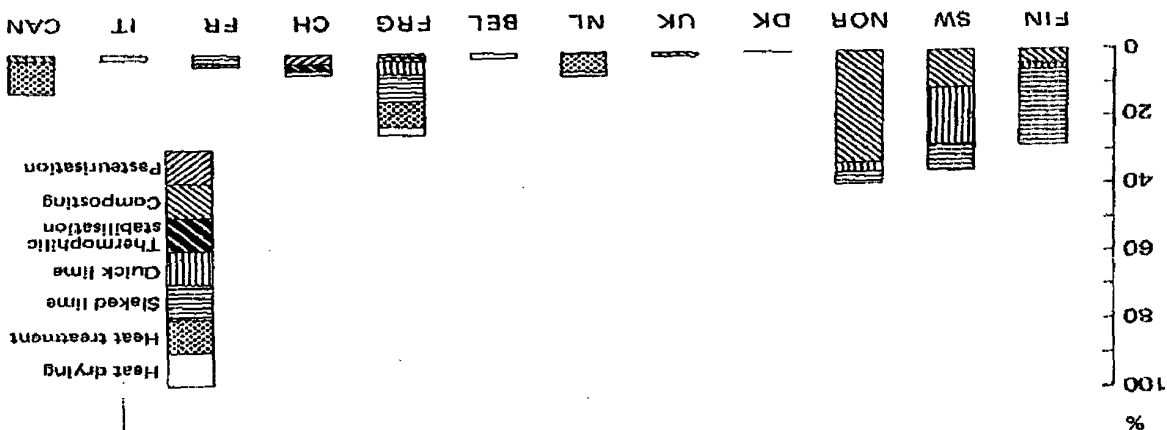
2.10 REFERENCES

[1] Breer, C., Hess, E. and Keller, U. (1979) Soll Klärschlamm vor oder nach dem Ausfaulen pasteurisiert werden? *Gas-Wasser-Abwasser* 59, 323–328.

[2] Keller, U. (1982) Experiences and development of the sludge pasteurization in Altenrhein. In: Bruce, A. M., Havelaar, A. H. and l'Hermite, P. (eds.) – *Disinfection of Sewage Sludge: Technical, Economic and Microbiological Aspects*. Proceedings of a Workshop held in Zurich May 11–13, 1982. D. Reidel Publishing Company, Dordrecht, pp. 53–67.

[3] Clements, R. P. L. (1982) Sludge hygienization by means of pasteurization prior to digestion. In: Bruce, A. M., Havelaar, A. H., and l'Hermite, P. (eds.) – *Disinfection of Sewage Sludge: Technical, Economic and Microbiological Aspects*. Proceedings of a Workshop held in Zurich May 11–13, 1982. D. Reidel Publishing Company, Dordrecht, pp. 37–52.

[4] Philipp, W. (1981) Vergleichende hygienische Untersuchungen über die Wirkung der Klärschlammpasteurisierung vor und nach der mesophilen, anaeroben, alkalischen Schlammfaulung Diss. Justus Liebig – Univ. Giessen.

[5] Strauch, D. and Berg. T. (1980) Mikrobiologische Untersuchungen zur Hygienisierung von Klärschlamm. 2. Versuche an Pasteurisierungsanlagen. *GWF-Wasser/Abwasser* 121, 184–187.

[6] Suess, A., Lessel, T., and Haisch, A. (1982) Technical and economical aspects of irradiation. In: Bruce, A. M., Havelaar, A. H, and l'Hermite, P. (eds.) – *Disinfection of Sewage Sludge: Technical Economic and Microbiological Aspects*. Proceedings of a Workshop held in Zurich May 11–13, 1982. D. Reidel Publishing Company, Dordrecht, pp. 179–190.

[7] Etzel, E., Born, G. S., Stein, J., Helbing, T. J., and Baney, G. (1969) Sewage sludge conditioning and disinfection by gamma irradiation. *Am. J. Public Health*, 59, 2067–2076.

[8] Sauer, J. (1969) Der Einfluss radioaktiver Bestrahlung auf die Lebensfähigkeit von Wurmeiern. *Wasser-Abwasser-Forschung* 2, 64–65.

[9] Brandon, J. R. (1976) Thermoradiation treats sewage sludge. *Water Sewage Works*. 123, 126–129.

[10] Lund, E., Lydholm, B., and Nielsen, A. L. (1982) The fate of viruses during sludge stabilization, especially during thermophilic digestion. In: Bruce, A. M.; Havelaar, A. H. and l'Hermite, P. (eds.) – *Disinfection of Sewage Sludge: Technical, Economic and Microbiological Aspects*. Proceedings of a Workshop held in Zurich May 11–13, 1982. D. Reidel Publishing Company, Dordrecht, pp. 114–124.

[11] Strauch, D. (1980) Mikrobiologische Untersuchungen zur Hygienisierung von Klärschlamm. 7. Weitere Untersuchungen an einem Verfahren zur aerob-thermophilen Schlammstabilisierung. *GWF-Wasser/Abwasser* 121, 552–555.

[12] Berg, G. and Berman, D. (1980) Destruction by anaerobic mesophilic and thermophilic digestion of viruses and indicator bacteria indigenous to domestic sludges. *Appl. Environ. Microbiol.* 39, 361–368.

[13] Kabrick, R. M. and Jewell, W. J. (1982) Fate of pathogens in thermophilic aerobic sludge digestion. *Water Res.* 16, 1051–1060.

[14] Parent, B. C. (1982) Composting – technical and economical aspects. In: Bruce, A. M., Havelaar, A. H. and l'Hermite, P. (eds.) – *Disinfection of Sewage Sludge: Technical, Economic and Microbiological Aspects*, Proceedings of a Workshop held in Zurich May 11–13, 1982. D. Reidel Publishing Company, Dordrecht, pp. 139–149.

[15] Strauch, D., Berg, T., and Fleischle, W. (1980) Mikrobiologische Untersuchungen zur Hygienisierung von Klärschlamm. 3. Versuche bei der Mietenkompostierung von Stroh mit Faulschlamm. *GWF-Wasser/Abwasser* 121, 298–301.

[16] Willson, G. B., Parr, J.F., Epstein, E., Marsh, P. B., Chaney, R. L., Colacicco, D., Burge, W. D., Sikora, L. J., Rester, C. F., und Hornick, S. (1980) *Manual for Composting Sewage Sludge by the Beltsville Aerated Pile Method*. US Env. Prot. Agency, Cincinnati, OH. EPA–600/8–80–022.

[17] Burge, W. D., Cramer, W. N., and Epstein, E. (1978) Destruction of pathogens in sewage sludge by composting. *Trans. ASEA.* 21, 510–514.

[18] Bertoldi, M.de, Coppola, S., and Spinosa, L. (1982) Health and economic implications in sewage-sludge composting. In: Bruce, A. M., Havelaar, A. H., and l'Hermite, P. (eds.) – *Disinfection of Sewage Sludge: Technical, Economic and Microbiological Aspects*. Proceedings of a workshop held in Zurich May 11–13, 1982. D. Reidel Publishing Company, Dordrecht, pp. 165–178.

[19] Stauch, D., Berg, T., and Fleischle, W. (1980) Mikrobiologische Untersuchungen zur Hygienisierung von Klärschlamm. 4. Untersuchungen an Bioreaktoren. *GWF-Wasser/Abwasser* 121, 331–334.

[20] Strauch, D., Berg, T., and Fleischle, W. (1980) Mikrobiologische Untersuchungen zur Hygienisierung von Klärschlamm. 5. Untersuchungen an Bio-Zellen-Reaktoren. *GWF-Wasser/Abwasser* 121, 392–394.

[21] Lofgren, L, Tullander, V., and Hovsenius, G. (1978) Occurrence and survival of micro-organisms in the combined composting of household waste and sludge. Statens Naturvardsverk. SNV PM 1077.

[22] Strauch, D. and Berg, T. (1980) Mikrobiologische Untersuchungen zur Hygienisierung von Klärschlamm. 6. Untersuchungen an einem Verfahren zur Schlammverfestigung mit Branntkalk. *GWF-Wasser/Abwasser* 121, 493–495.

[23] Koch, K. and Strauch, D. (1981) Inaktivierung von Polio- und Parvovirus im Klärschlamm durch Kalkbehandlung. *Zbl. Bakt. Hyg. I Abt. Org. B* 174, 335–347.

CHAPTER 3

Stabilisation and disinfection - their relevance to agricultural utilisation of sludge

E. B. Pike and R. D. Davis

3.1 INTRODUCTION

Stabilisation of raw sludge is carried out primarily to reduce odour and its content of organic matter and dry solids, rendering it easier to handle and transport. During the anaerobic processes of digestion, storage and lagooning, microbiological activity brings about hydrolysis of cellulose, fats and proteinaceous materials in the sludge resulting in the production of fatty acids and amino acids as intermediates, followed by release of carbon as gaseous carbon dioxide and methane and by release of ammonium ion. During aerobic stabilisation, similar hydrolytic activities occur but the presence of oxygen results in more complete oxidation to carbon dioxide. However, much of the residual organic solids are composed of microbial cells, which are inherently resistant to degradation. A sludge may therefore conveniently be regarded as stabilised by these methods when roughly 40 per cent of the volatile solids have been oxidised. A subsidiary result is that the content of potentially toxic elements in the dry solids increases during biological stabilisation.

Stabilisation may also be achieved chemically by addition of lime or other chemicals to inhibit microbiological activity, thereby preventing the release of odorous compounds.

It is one of the objects of this chapter to show how stabilisation brings about two major additional benefits, the decay of disease-causing micro-organisms and, in the case of anaerobic digestion, the more rapid availability of the nitrogen content for crops. Stabilisation thus increases the manurial value of sludge and acts as a partial barrier to the transmission of disease, so that it can be spread on land with less restriction than can raw sludge.

On the other hand the main aim of disinfection is to render sludge free from infection, so that it can be spread with fewest restrictions.

The various effects of stabilisation and disinfection will now be considered.

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[24] Polprasert, C. and Valencia, L. G. (1981) The inactivation of faecal coliforms and *Ascaris ova* in faeces by lime. *Water Res.* 15, 31–36.

[25] Strauch. D. and Schreiber-Rothschild. R. (1980) Mikrobiologische Untersuchungen zur Hygienisierung von Klarschlamm. 8. Der Einfluss von Kalk als Konditionierungsmittel bei Kammerfilterpressen. *GWF-Wasser/ Abwasser* 121, 607–611.

[26] Havelaar, A. H. and Bruce, A. M. (1982) Disinfection of sewage sludge. An enquiry among member states of the EEC and some other countries. In: Bruce, A. M.; Havelaar, A. H. and l'Hermite, P. (eds.) — *Disinfection of Sewage Sludge: Technical, Economic and Microbiological Aspects.* Proceedings of a Workshop held in Zurich May 11–13, 1982. D. Reidel Publishing Company, Dordrecht, pp. 245–252.

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Fig. 1.13 Death rate curve.

Fig. 1.11 Thermal wheel (Courtesy of the Electricity Council.)
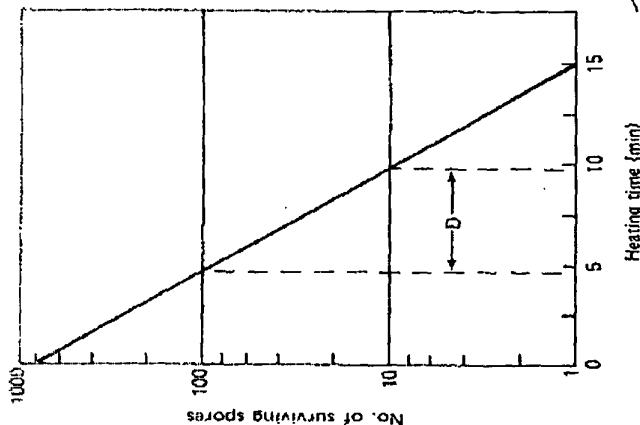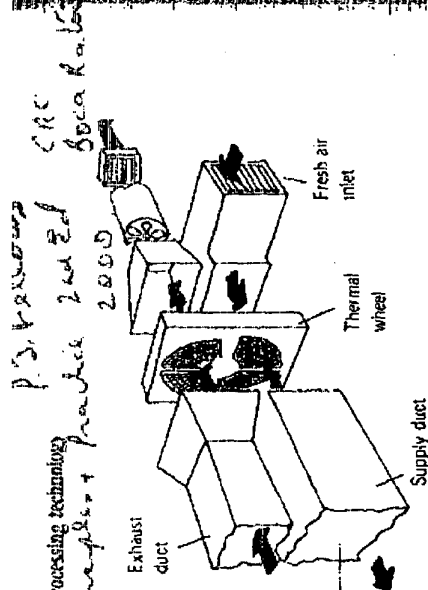

Fig 1.12 Rotaire wheel (Courtesy of the Electricity Council.)

Table 1.11 Thermal properties of selected nutritional and sensory components of foods in relation to heat-resistant enzymes and bacteria

Component	Source	pH	z (°C)	D_{121} (min)	Temperature range (°C)
Thiamin	Carrot purée	5.9	25	158	109–149
Thiamin	Pea purée	Natural	27	247	121–138
Thiamin	Lamb purée	6.2	25	120	109–149
Lysine	Soya bean meal	–	21	786	100–127
Chlorophyll a	Spinach	6.5	51	13.0	127–149
Chlorophyll a	Spinach	Natural	45	34.1	100–130
Chlorophyll b	Spinach	5.5	79	14.7	127–149
Chlorophyll b	Spinach	Natural	59	48	100–130
Anthocyanin	Grape juice	Natural	23.2	17.8*	20–121
Betanin	Beetroot juice	5.0	58.9	46.6*	50–100
Carotenoids	Paprika	Natural	18.9	0.038*	52–65
Peroxidase	Peas	Natural	37.2	3.0	110–138
Peroxidase	Various	–	28–44	–	–
Clostridium botulinum spores type A + B	Various	>4.5	5.5–10	0.1–0.3*	104
Bacillus stearothermophilus	Various			10.5*	

confectionery is described by Anon. (1983a, b, c). The use of air knives for package drying is described by Anon. (1983d) and Beevers (1985). Other energy-saving techniques during dehydration are described by Senhaji and Bimbenet (1984), Flink (1977) and Green (1982).

1.4.5 Effect of heat on micro-organisms

The preservative effect of heat processing is due to the denaturation of proteins, which destroys enzyme activity and enzyme-controlled metabolism in micro-organisms. The rate of destruction is a first-order reaction; that is when food is heated to a temperature that is high enough to destroy contaminating micro-organisms, the same percentage die in a given time interval regardless of the numbers present initially. This is known as the *logarithmic order of death* and is described by a *death rate curve* (Fig. 1.13)

The time needed to destroy 90% of the micro-organisms (to reduce their numbers by a factor of 10) is referred to as the *decimal reduction time* or *D value* (5 min in Fig. 1.13). It

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There are two important implications arising from the decimal reduction time: first, the higher the number of micro-organisms present in a raw material, the longer it takes to reduce the numbers to a specified level. In commercial operation the number of micro-organisms varies in each batch of raw material, but it is difficult to recalculate process times for each batch of food. A specific temperature–time combination is therefore used to process every batch of a particular product, and adequate preparation procedures (Chapter 3) are used to ensure that the raw material has a satisfactory and uniform microbiological quality. Second, because microbial destruction takes place logarithmically, it is theoretically possible to destroy all cells only after heating for an infinite time. Processing therefore aims to reduce the number of surviving micro-organisms by a pre-determined amount. This gives rise to the concept of *commercial sterility*, which is discussed further in Chapters 10–12.

The destruction of micro-organisms is temperature dependent; cells die more rapidly at higher temperatures. By collating D values at different temperatures, a *thermal death time* (TDT) curve is constructed (Fig. 1.14). The slope of the TDT curve is termed the z value and is defined as the number of degrees Celsius required to bring about a ten-fold change in decimal reduction time (10.5°C in Fig. 1.14). The D value and z value are used to characterise the heat resistance of a micro-organism and its temperature dependence respectively.

There are a large number of factors which determine the heat resistance of micro-organisms, but general statements of the effect of a given variable on heat resistance are not always possible. The following factors are known to be important.

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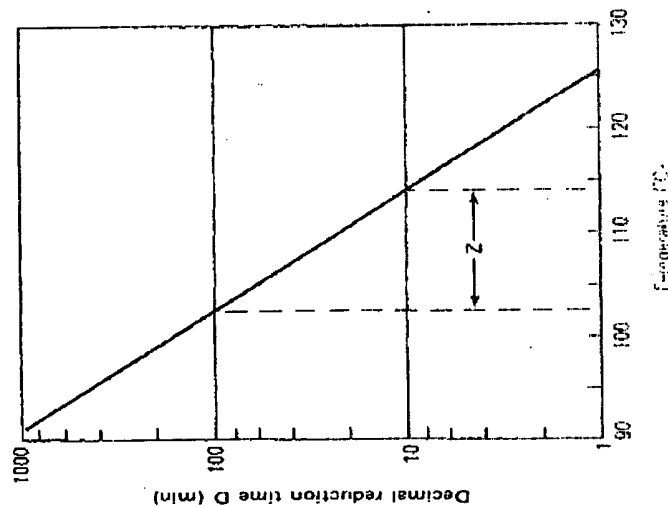
1. *Type of micro-organism.* Different species and strains show wide variation in their heat resistance (Table 1.11). Spores are much more heat resistant than vegetative cells.

2. *Incubation conditions* during cell growth or spore formation. These include:
    (a) temperature (spores produced at higher temperatures are more resistant than those produced at lower temperatures)
    (b) age of the culture (the stage of growth of vegetative cells affects their heat resistance)
    (c) culture medium used (for example mineral salts and fatty acids influence the heat resistance of spores).

3. *Conditions during heat treatment.* The important conditions are:
    (a) pH of the food (pathogenic and spoilage bacteria are more heat resistant near to neutrality; yeasts and fungi are able to tolerate more acidic conditions but are less heat resistant than bacterial spores)
    (b) water activity of the food (section 1.5) influences the heat resistance of vegetative cells; in addition moist heat is more effective than dry heat for spore destruction
    (c) composition of the food (proteins, fats and high concentration of sucrose increase the heat resistance of micro-organisms; the low concentration of sodium chloride used in most foods does not have a significant effect; the physical state of the food, particularly the presence of colloids, affects the heat resistance of vegetative cells)
    (d) the growth media and incubation conditions, used to assess recovery of micro-organisms in heat resistance studies, affect the number of survivors observed.

Most enzymes have D and z values within a similar range to micro-organisms, and are therefore inactivated during normal heat processing. However, some enzymes are very heat resistant. These are particularly important in acidic foods, where they may not be completely denatured by the relatively short heat treatments and lower temperatures required for microbial destruction. The factors which influence heat resistance of enzymes are similar to those described for micro-organisms and are discussed in detail by Whitaker (1972).

A knowledge of the heat resistance of the enzymes and/or micro-organisms found in a specific food is used to calculate the heating conditions needed for their destruction. In practice the most heat resistant enzyme or micro-organism likely to be present in a given food is used as a basis for calculating process conditions. It is assumed that other less heat-resistant species are also destroyed. Methods for the calculation of processing time are described in Chapter 12.

1.4.6   Effect of heat on nutritional and sensory characteristics

The destruction of many vitamins, aroma compounds and pigments by heat follows a similar first-order reaction to microbial destruction. Examples of D and z values of selected vitamins and pigments are shown in Table 1.11. In general both values are higher than those of micro-organisms and enzymes. As a result, nutritional and sensory properties are better retained by the use of higher temperatures and shorter times during heat processing. It is therefore possible to select particular time-temperature combinations from

Decimal reduction time D (min) — 1000, 100, 10, 1
z
Temperature (°C) — 90, 100, 110, 120, 130